

REMARKS

Claims 1-61 are pending herein, of which claims 16-61 have been withdrawn as being directed to a non-elected invention.

1. Claim 5 was rejected under §112, second paragraph. Claim 5 has been amended herein to remove this rejection.

2. Claims 1-15 were rejected under §103 over JP '899 (Takahashi et al.). This rejection is respectfully traversed for the following reasons.

The claimed invention (claim 1) is directed to a large-sized sapphire single crystal, comprising a single crystal sheet having a width not less than 15 cm, a length greater than the width and a thickness not less than 0.5 cm. Independent claim 6 is also directed to a sapphire single crystal comprising a single crystal sheet, and recites that the sheet has a variation of thickness of not greater than 0.2 cm. Claim 11 combines the features of claims 1 and 6, and claim 12 recites an as-grown single crystal sheet, including a main body portion and a neck portion. The neck portion has a uniform construction as quantified by Δ_T as recited.

The claimed invention provides for large-sized sapphire single crystals in the form of single crystal sheets, and represents a notable advancement over the state of the art. In this respect, the attention of the PTO is drawn to the previously submitted technical article entitled "Large Diameter Sapphire Window From Single Crystal Sheets" from the Proceedings of the 5th DOD Electromagnetic Window Symposium. The article describes an attempt to scale-up EFG-grown sapphire sheets to dimensions of 30.5 cm wide X 48 cm long X 0.25 cm thick. Although the article describes formation of such a sized single crystal sapphire sheet, the technology described therein is limited, particularly confined to formation of what is essentially medium-sized sapphire single crystal sheets.

The claimed invention provides sapphire single-crystal sheets having larger dimensions and/or superior uniformity over the sheets achieved in the article. In this respect, Applicants have developed a process flow incorporating notable features, such as use of a high aspect ratio crucible (see FIG. 4, paragraph 28 of the present specification), incorporation and manipulation

of a gradient trim system for dynamically adjusting the thermal gradient along the length of the die through which the sheet is formed (see FIGs. 1-2, gradient trim system 50, paragraphs 25 and 36, for example), incorporation of a particular heat shield assembly providing a baseline thermal profile (see shield assembly 26, FIGs. 1-3 and paragraphs 21 and 26), as well as other structural and process-oriented features. In a nutshell, Applicants have developed enabling technology to provide next-generation large-sized single crystal sapphire sheets by EFG, having a high level of uniformity.

Turning to Takahashi et al., the attention of the PTO is directed to the English-language translation thereof, enclosed herewith. A review of Takahashi et al. reveals that this reference is no more relevant than the state of the art over which the claimed invention has been developed, and the most pertinent disclosure appears to be provided in connection with Example 3 describing a sapphire mono-crystal block having the dimensions of 10 cm X 10 cm X 3 cm. Certainly, the single crystal block of Takahashi et al. does not meet the geometric features of the claimed invention. In this respect, the PTO has argued that it would have been obvious to one of ordinary skill in the art to modify the dimensions of Takahashi et al. to meet those presently claimed. However, Takahashi et al. do not even remotely enable such a modification. As described above, multiple process and structural innovations have been developed by the Applicants to enable formation of large-sized and uniform single crystal sapphire sheets. Clearly, there is no teaching or remote suggestion of high aspect crucibles, use of a stationary shield structure to provide a baseline thermal profile, utilization of a radiant trim system, as well as other features described throughout the present specification, or any alternative technology to achieve the claimed invention. Accordingly, it is quite clear that Takahashi et al. fail to disclose or even remotely suggest all features of the claimed invention.

For at least the foregoing reasons, Applicants respectfully submit that the presently claimed invention would not have been obvious over Takahashi et al. Accordingly, reconsideration and withdrawal of the §103 rejection are respectfully requested.

Applicants respectfully submit that the present application is now in condition for allowance. Accordingly, the Examiner is requested to issue a Notice of Allowance for all pending claims.

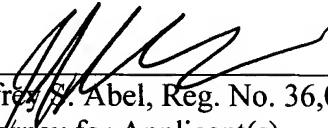
Should the Examiner deem that any further action by the Applicants would be desirable for placing this application in even better condition for issue, the Examiner is requested to contact Applicants' undersigned attorney at the number listed below.

Applicants does not believe that any additional fees are due, but if the Commissioner believes additional fees are due, the Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment, to Deposit Account Number 50-2469.

Respectfully submitted,

Date

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OPEN PATENT OFFICIAL REPORT

Deformation Reform Method of Sapphire Monocrystal Sheet

Patent Number: S55-173669

Date: December 9, 1980

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Inventor: Kenichi Masukawa, Toshiba Ceramic

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Description

1. Name of Invention

Deformation reform method of sapphire monocrystal sheet

2. Range of patent claim

Sapphire monocrystal sheet heat treatment at under the temperature between 1150 and 1400°C deformation reform method.

3. Description of invention

This invention regards the deformation reform method of the sapphire monocrystal sheet.

Recently, rearing technology of the sapphire monocrystal has been progressed, and sapphire monocrystal becomes large-sized with superior quality. Therefore, the sapphire monocrystal sheet has been used in electronic industrial field such as

semiconductor industry and the optical industrial field. For example, substrate for 808, substrate for the integrated circuit, the window board for the ultraviolet ray and the plate for the infrared ray, etc. As for these thin plates should be high accuracy for the flatness, degree of parallelization, thickness and crystal orientation, because using in optical systems or electron optics systems.

By the way, the former sapphire monocrystal sheet manufacturing process was cutting off, processing, furthermore grinding, or lapping or polishing. After the grinding or after the surface processing, the thin plate has the sled between 4 and 7 μ m/cm. The sapphire monocrystal sheet with this type of sled has various problems for usage. For example, 808 substrates make out of sapphire monocrystal sheet, and then multiple high accuracy elements will form on silicon layer of the 808 substrate. The 808 substrate should have 25 μ m or less flatness and degree of parallelization must be 7.5 μ m or less. If the 808 substrate has great sled such as mentioned above, it becomes hindrance in element formation of high accuracy.

Currently, the sled of the above-mentioned sapphire monocrystal sheet will be fixed by surface process technology. However, this technology has several problems such as required skillful technician, time consuming, and low yield rate.

Compare with the old technology, the inventors of this patent found the solution of greatly reducing the sled on the sapphire monocrystal by cutting off, grinding or furthermore the sapphire monocrystal thin plate which surface has been processed, will be treated by heat between 1150 and 1400 °C. The mechanism is not clear where the sled of the sheet is reformed by this kind of heat treatment, but, according to the research of these inventors it is thought the following reason. The sapphire monocrystal was cut or

grind or surface process, and generate the sapphire monocrystal thin sheet. The sapphire monocrystal thin sheet has been stressed and quarried out, these become the goblet and the sled occurs in the sheet. If the thin plate has heat treatment under specified temperature, the goblet inside the sheet being disappeared, it tries to return to original form and the sled is reformed.

Namely, features of this invention is heat treatment of sapphire monocrystal sheet under the temperature between 1150 and 1400°C.

The sapphire monocrystal sheet in this invention means; the sapphire monocrystal sheet obtain after cut and grind of the sapphire monocrystal. And the cut and grind sapphire monocrystal sheet obtain after surface process treatment such as lapping and polishing.

The reason of restricted heat treatment temperature above is; when the temperature is under 1150°C, the sled revision effect is not sufficient. When the temperature exceeds 1400 °C, the sapphire monocrystal sheet has remarkable impurity spread and pollution, the other wise; there is no reform effect above 1400°C and disadvantageous economical efficiency.

Furthermore, the annealing was executed previously. The annealing removes strain that gains at crystallizing and remains inside of crystal. During annealing, the sapphire monocrystal was slowly cooling down less than 200°C/hour speed after heated with 1800 - 1900°C , in 5 – 10 hours. However, this method required high temperature such as 1800 - 1900°C with 5-10 hours long heating process, and slow cooling speed 200°C/hour. The heating process in this invention is different. In fact, applying heat

treatment of this invention to the above-mentioned annealing, it cannot achieve removing the sufficient strain.

Next, execution example of this invention is explained.

Execution Example 1

First, the disk for 808 substrate was quarried out from the cylinder shape sapphire monocrystal ingot of 76mm diameter and 30mm thickness. This disk thickness was 0.76mm average, thickness difference inside the disk (Δt) was 0.076mm average, the cut surface sled quantity was 0.089mm average. Second, the disk attached in the both sides grinder, ground from both sides and producing grinded disk with 0.06mm average thickness, 0.015mm even thickness board, and 0.012mm even sled quantity. Third, 5 grinded disks mentioned above each plate total 20 attached to 4 iron-made plates with 230mm diameter and 3 μ m flatness, then one side of those disk was ground with the surface grinding machine. After the grinding, each disk is still attached, their average flatness are 4 μ m.

Next, 10 disks peel off from 2 iron-made plates which mentioned above, 10 disks were stuck to other 2 plates that way. 10 peeled off disks from 2 iron-made plates put on an alumina-quality board one each flatly, then the alumina-quality board insert in the electric furnace and heat to 1250°C in the atmosphere, keeping at the temperature for 1 hour for heat treatment after loading this alumina-quality board to the alumina-quality core tube. After that, the electrification to the furnace been stopped, then the disk has removed from the furnace when disk is cool down to room temperature. 10 disks, which

mentioned above attach to 2 iron-made plates again. Heat treated disks as mentioned above and non-heat treated disk which glued together has been grounded simultaneously until the surface discontinuity completely disappeared. Then, all disks were peeled off, specified cleaning was done. Thickness of each disk, all board thickness differences (Δt), and sled quantity was measured. The following table 1 is the result of measurement.

Table 1

<u>Heat Treated Disks</u>				<u>Non Heat Treated Disks</u>			
<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>	<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>
A-1	485 μm	13 μm	12 μm	B-1	524 μm	15 μm	150 μm
A-2	483	12	10	B-2	521	18	170
A-3	483	12	13	B-3	525	16	150
A-4	476	14	14	B-4	531	15	180
A-5	482	10	11	B-5	535	19	190
A-6	476	7	12	B-6	506	14	130
A-7	474	10	11	B-7	508	13	170
A-8	479	11	9	B-8	507	16	150
A-9	473	8	13	B-9	507	14	160
A-10	476	9	10	B-10	505	17	180

Note: A/T means Average Thickness

T/D means All Board Thickness Differences

S/Q means Sled Quantity

It is clear from the above-mentioned Table 1, the all board thickness differences (Δt), and the sled quantity both are decreased effectively on heat-treated sapphire monocrystal disk in comparison with the non heat-treated sapphire disk.

Execution Example 2

The disk of 76mm diameter was scooped out from processed sapphire monocrystal material with 80mm width and 1mm thickness, which were reared in ribbon base. The disk was installed in the both sides grinder, then it will be ground from both sides until the disk will be 568 μm average thickness, 11 μm average thickness difference and 8 μm average sled quantity. Next, the 5 grinded disks mentioned above for each plate total 10 disks attached to 2 iron-made plates of 230 μm diameter and 3 μm flatness, then one side of those disks was ground by surface grinding machine. After grinded, average flatness of each disk was 3 μm according to our measurement while they attached together. Those disks's ground surface was continuously ground by using diamond abrasive grain.

Next, 5 disks were peeled from 1 iron-made plate above mentioned, another 5 disks left on another plate as stuck together. The peeled 5 disks were leaned against an

alumina-quality board at specified interval at a time. The board insert to an electric furnace after loading to the alumina-quality core tube. Then, the board heated up to 1150°C with increasing temperature rate 400 °C/hour at the atmosphere, and kept the temperature for three hours. After that, to stop the electrification to the furnace, then the disks were removed from the furnace when cooling to room temperature. Again, the 5 disks attached an iron-made plate mentioned above. The heat-treated disks as mentioned above and non-heat treated disk which glued together has been chemically polished simultaneously until the surface discontinuity completely disappeared. Then, all disks were peeled off from the plate and specified cleaning was done. Thickness of each disk, all board thickness differences (Δt), and sled quantity was measured. The following table 2 is the result of measurement.

Table 2

<u>Heat Treated Disks</u>				<u>Non Heat Treated Disks</u>			
<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>	<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>
C-1	490 μm	12 μm	8 μm	D-1	508 μm	14 μm	170 μm
C-2	492	10	11	D-2	507	17	150
C-3	490	8	5	D-3	506	20	190
C-4	493	9	7	D-4	509	18	200
C-5	495	8	12	D-5	507	19	180

Note: A/T means Average Thickness

T/D means All Board Thickness Differences

S/Q means Sled Quantity

Execution Example 3

The square plate of 1.8mm thickness cut out from the sapphire monocrystal block which size of 100mm (Length) x 100mm(Width) x 30mm (Tall). The square plate's average thickness is 1.83mm, an average thickness difference is 0.097mm and an even sled quantity is 0.093mm. Next, the 4 square plates mentioned above each plate total 8 square plates attached to 2 high density alumina plates of 230mm diameter and 3 μ m flatness, then, one side of those square plate was ground by surface grinding machine. After grinding, each square plate's average flatness was 3 μ m according to our measurement while those square plates were attach together. Those disks's ground surface was continuously ground until scratch and the bit on the grinded surface completely disappear. Furthermore, each squares plate peeled off from the alumina plate, and then square plates turned over and attached to the same alumina plate. After that, the square plates were ground by grinding machine, then an average thickness of square plate is 1.648 mm and an average flatness of square plate is 3 μ m according to our measurement. After that, the square plates have been grounded again until scratch and bit disappear completely.

Next, 4 square plates were peeled from one alumina plate mentioned above, and other 4 square plates were stuck to another alumina plate as way it is. The 4 peeled square plates were cleaned, and then learned against an alumina quality board with specified interval each other, then the alumina-quality board with 4 square plates insert to an alumina-quality furnace which connected with an oxygen supply tube. Then the alumina-quality board with 4 square plates insert to an electric furnace which equipped SIC heating element then, increased temperature up to 1300 °C with 300 °C/hour speed in the oxygen gas atmosphere, and the temperature was kept around 30 minutes. After that, the electrification to the furnace stopped, then the square plates were removed from the furnace when cooling to room temperature in oxygen gas atmosphere. And, those 4 square plates attached to one alumina plate, which mentioned above again. Thickness, all board thickness differences (Δt), and sled quantity of both heat treated plate and non heart treated plate was measured. The following table 3 is the result of measurement.

Table 2

<u>Heat Treated Disks</u>				<u>Non Heat Treated Disks</u>			
<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>	<u>Sample</u>	<u>A/T</u>	<u>T/D</u>	<u>S/Q</u>
E-1	1.610 μm	14 μm	5 μm	F-1	1.608 μm	13 μm	14 μm
E-2	1.610	10	5	F-2	1.607	11	10
E-3	1.608	7	4	F-3	1.609	12	15
E-4	1.610	9	5	F-4	1.610	13	17

Note: A/T means Average Thickness

T/D means All Board Thickness Differences

S/Q means Sled Quantity

Described above, according to this invention, the sled of sapphire monocrystal dramatically can adjust with the following process. Giving surface treatment after cutting and grinding, and then processed by heat with between 1150-1400 °C. Therefore, the sapphire monocrystal sheets can be utilized in electronics industry such as semiconductor industry and optical industry.